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Technical Note N-659

INVESTIGATION OF POSSIBLE METHODS FOR TESTING GAS FILTERS

BY

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16 November 1964

U. S. NAVAL CIVIL ENGINEERING LABORATORY
Port Hueneme, California

INVESTIGATION OF POSSIBLE METHODS FOR TESTING GAS FILTERS

Y-F011-08-03-301

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ABSTRACT

An investigation and literature search was made of activated charcoal, mercaptans and the use of odors in testing gas penetration of carbon filters. Tests were performed on the detection of mercaptans, especially N-amyl mercaptan. This work was done in connection with Task Y-F011-08-03-301, "Development of Test Procedure and Kit for Proof Testing Installed CBR Filter Systems," and is discussed in this technical note.

It was found that the idea of using an odor to test filters may have merit, but mercaptans appear too hazardous for this purpose. There are, however, other sources of odor that can be investigated. Using human sense of smell for detecting gas leaks may be practical, but it is not as easy to do as it might appear. Most of the more quantitative methods that are being used at the present time lack sensitivity, but gas chromatography is worthy of further consideration.

INTRODUCTION

The purpose of Task Y-F011-08-03-301 is to develop a field kit to test installed CBR collective protectors. There are two parts to the task: (1) testing the particulate filter for particle penetration, and (2) testing the gas filter for gas penetration. The work discussed here is a contribution to part two.

It is required that the kit be capable of testing the gas filter for leaks and, if possible, for residual service life. Residual service life is the life left in a filter after it has aged and has been exposed to gases which are adsorbed by the carbon.

The gas filter in the collective protector is made from an adsorbent bed of ASC activated charcoal.[1] Activated charcoal selectively adsorbs certain gases and lets others pass through unaffected. War gases are adsorbed to a high degree, and other gases, including freons and mercaptans, are also adsorbed in varying degrees.

This report is divided into two sections. The first discusses activated carbon and is based on both a literature study and the opinions of the author. The second discusses odor detection and is based on experimental work with mercaptans suggested by a literature study.

ACTIVATED CARBON

Carbon Characteristics

Activated carbon is a granular solid manufactured from organic matter such as cocoanut shells, and is made extremely porous in order to create a very large surface area. There are two basic methods by which the carbon adsorbs gases: physical adsorption and chemical adsorption. In physical adsorption, gas molecules appear to be held to the carbon by weak intermolecular attraction. In chemical adsorption, the gaseous molecules react chemically at the surface of the carbon. Neither of these processes is clearly understood.

The ability of carbon to adsorb depends on the nature of the activation process so that similar grades of carbon from different manufacturers have different adsorption properties. In a given batch of activated carbon, the adsorption characteristics will vary with the type of gas, and with the temperature, humidity, and pressure. With a mixture of gases the adsorption characteristics will also vary with the types of gases and their concentrations.[2]

An adsorption study of various hydrocarbon gas mixtures indicated that one will usually be adsorbed in preference to the others,[3] and the gas preferentially adsorbed will be the one for which the carbon has the highest capacity when the gases are adsorbed separately. In a homologous series of hydrocarbons, carbon has a tendency to adsorb larger amounts of those gases with the highest molecular weight. Less of a given hydrocarbon is adsorbed from a mixture than would be adsorbed from the pure gas under the same conditions. This is shown by the following equation:

$$\sum_{i=1}^{n} \frac{N_i}{N_i'} = 1$$

where: N_i = quantity of gas i adsorbed from a mixture

N_i' = quantity of gas i adsorbed when gas i is pure.

Note that these conclusions are based on a study of related hydrocarbon gases. There is no assurance that compounds with functional groups, such as mercaptans, will behave as the hydrocarbons do, but it is possible that they will.

Experimental Test Criterion

The government uses gas mask canisters when it tests ASC charcoal.[1] These canisters are tested with chloropicrin, hydrocyanic acid, phosgene, cyanogen chloride, and arsine, using the All Purpose Gas Life Testing Apparatus Q-2. The flow rates of the test gases are either 32 liters per minute constant flow or 50 liters per minute intermittent flow. The concentrations of each gas, which the canisters must remove from the air stream for a specified period of time (20 to 45 minutes), range from 4 to 50 mg per liter. Fifty mg per liter of chloropicrin corresponds to about 1400 parts per million (ppm) by volume. According to Hassler,[2] the charcoals which are best for filtering chloropicrin are best for filtering most of the other war gases, so that, lacking a better criterion, the one for testing with chloropicrin might be considered for testing with non-toxic gases. Thus, it was decided to test the filter with a gas concentration of 1400 ppm, and until a better criterion is found, consider concentrations greater than 1 ppm downstream from the filter as indicative of failure.

Testing Carbon Beds for Leaks

When a new chemical is being investigated for testing carbon beds, it is necessary to determine the effects of the chemical on the carbon as well as to determine the effectiveness of the carbon in adsorbing the chemical. Many gases which are physically adsorbed can, to some extent, be desorbed by passing clean air through the carbon. However, gases such as hydrogen sulphide, react chemically on the carbon and prevent desorption. They may also reduce the carbon's ability to adsorb other gases.[4]

The following procedure was designed for finding a non-toxic gas suitable for testing installed carbon filters. It has not been tried as yet. Two separate carbon beds are required and they are first weighed. Then a mixture of air and the selected war gas is drawn through one of the beds until a trace of the gas is detectable. The time necessary for penetration is measured, and the bed is again weighed to determine the weight of war gas adsorbed.

After the ability of the carbon to adsorb the war gas has been determined, the test gas candidate is adsorbed in the other carbon bed using the procedure just described and weighing the carbon before and after the test. Clean air is then passed through this bed to desorb the test gas. By weighing the bed periodically, one can tell when the test gas has been desorbed. If the test gas appears to be satisfactory the second bed should then be tested with the war gas as was done with the first bed and the two compared to see if the ability of the carbon to adsorb the war gas has been reduced. Finally, a similar test should be made to determine if water vapor affects the adsorptive capacity of the carbon adversely by reducing the adsorption of the test gas candidate.

Testing Carbon Beds for Residual Life

Testing for leaks was the primary purpose of the investigations described in this report, but it was also hoped that residual service life could be determined. A small, removable carbon filter can be placed in the air-stream near the main filter so that it will adsorb a portion of the gas. By measuring the increase in weight of the removable filter an estimate of the amount of gas adsorbed by the main filter can be obtained, and the remaining service life estimated. A chemical analysis of the carbon in the removable filter may give a more precise determination of the residual service life.

ODOR DETECTION AND THE USE OF MERCAPTANS

Discussion

It became evident that neither a suitable gas nor a method of detection is yet available for testing collective protectors in the field. Dr. James A. Young of the Naval Research Laboratory suggested that since the human nose is very sensitive to odors, one of the mercaptans might make a satisfactory test agent. Mercaptans are sulfur-hydrogen compounds with distinctive odors. If the mercaptan is introduced upstream, it should be adsorbed as it passes through the filter unless there is a leak, in which case the odor should be immediately evident to observers downstream.

Techniques of Odor Measurements

If filters are to be tested by using people to detect odors, it is necessary to understand some basic concepts of odor measurement. Much work on techniques for measuring the sensitivity of individuals to odors is described in the literature. Usually water solutions of the chemical under test are prepared in various concentrations. Each individual then sniffs the solutions in turn until a solution is found whose odor can just be detected. The concentration of this solution is the threshold odor concentration for the individual. A group of 10-40 persons often participate in this testing session, and the results are analyzed statistically. Since the sensitivity of the human nose to odors decreases logarithmically with the length of exposure, the testing sessions are necessarily brief.

It was found that odor sensitivities vary as much as a thousand-fold among individuals so that it is necessary to use a number of trained observers and to make several tests in order to determine threshold odors with reliability. Background odors confuse observers; when sniffing for a given odor, a similar odor will hinder detection, but a different one may improve it. Tobacco smoking and colds also change an observer's sensitivity.

Description of Mercaptans

Mercaptans are organic derivatives of hydrogen sulfide. They can be recognized in a chemical formula by the $(SH)^-$ group attached to a carbon atom in an organic molecule. Mercaptans are slightly acid[5] and are characterized by irritating, penetrating odors. They are usually flammable, explosive, or toxic. Table I[6,7,8] contains a list of some of the mercaptans, together with a few of their properties.

Table I. Properties of low boiling point mercaptans.

Mercaptan	Safe industrial limit of conc. in parts per million[6]	Hazardous properties[7]	Handling[7]	Boiling point °C	Flash[7] point °C	Explosive limits %	Remarks
Methyl	50	Flammable, irritant	Respirator or chemical goggles required for large or unknown concentrations.	7.6	14.2		
Ethyl	250	Explosive; fire hazard; affects nervous system. Strong odor but toxicity low.	Chemical safety goggles.	34.7		2.8-18.2	Odorant in liquefied petroleum gas[8]
n-Butyl	10	Very toxic; strong odor; military poison gas.	Adequate safety equipment required at all times including goggles, respirator.	98			

Blank spaces indicate no information was found.

Table I. Properties of low boiling point mercaptans. (continued)

Mercaptan	Safe industrial limit of conc. in parts per million[6]	Hazardous properties[7]	Handling[7]	Boiling Point °C	Flash[7] point °C	Explosive limits %	Remarks
Allyl Merc		No information but other allyl compounds <u>extremely</u> toxic.		67			
n-Amyl		Very flammable; irritant to mucous membranes.		126	18.3		Odorant in natural gas.[8]
Perchloromethyl	0.1			149			
n-Propyl				67			

Blank spaces indicate no information was found.

Mercaptan Detection Methods

Since the sensitivity of the human nose varies widely from person to person and with the length of exposure, a more quantitative measure of the mercaptan concentration is often desirable. Because of interest by the petroleum and pulp industries, a great deal of work has been done on separating and measuring mercaptans in industrial processes. However, there are few methods of measurement sufficiently sensitive for testing filters. Among the methods commonly used are gas chromatography, spot tests, colorimetric methods and titrations. Time limitations and lack of available information prevented investigating colorimetric methods and titrations, but the other two methods are discussed in this section.

References in the literature indicate that very promising results have been obtained with gas chromotography,[9] and that sensitive and accurate equipment may have been used for purposes similar to those of this task. One reference indicated that work has been done by Czerwonka, Boyland, and Gonzales of American Air Filter Company in Louisville, Kentucky. These men made practical application of the gas chromatograph to the evaluation of adsorptive beds and have been able to measure quickly the adsorptive power, retentivity, and breakdown time. Their work also suggested the possibility of making rapid field tests.[10] A cursory investigation was made to learn more about the work done at American Air Filter Company. It was found that Mr. Czerwonka had presented a paper on odor control, theory and measurement which, along with others, is to be published by the New York Academy of Sciences, but so far, these papers have not been published.

After considering the expense of using gas chromatography, it was decided to investigate some of the spot tests for detecting mercaptans. Spot tests employ various reagents to determine the presence or absence of a compound by using drop-sized samples. They are probably the simplest but also the least quantitative of the methods for measuring mercaptans.

Work with N-Amyl Mercaptan

N-amyl mercaptan was chosen to be tested first because it is an odorant for natural gas.[11] Sample solutions of n-amyl mercaptan in both ethanol and ether were prepared in concentrations of 1 ppm and of 1000 ppm. The following spot tests were then made:

a. Sodium Nitroprusside Test: One drop of mercaptan solution was added to two milliliters of a 1 per cent sodium nitroprusside solution. Then, when three drops of a 10 per cent sodium hydroxide solution are added, a wine color should result if a mercaptan is present. The wine color was obtained for the 1000 ppm sample but not for the 1 ppm sample.

b. Iodine-Azide Test: Three grams of sodium nitride and 100 ml of 0.1 N iodine solution were mixed. One drop of the mixture was placed on a watch glass, and one drop of mercaptan solution was added. Nitrogen bubbles should result when the mercaptan catalyzes the reaction between sodium nitride and iodine. The bubbles did form with the 1000 ppm solution of mercaptan, but very small, slowly evolving bubbles only were observed for the 1 ppm solution.

c. Cuprous Salts Test: Two solutions, A and B, were prepared as follows:

A. 1.5 g of cupric chloride and ammonium chloride were dissolved in water. To this solution were added 3 ml of concentrated ammonium hydroxide and enough water to make 50 ml.

B. Hydroxylamine hydrochloride was dissolved in water to make a 20 per cent solution.

For the test, equal volumes of solutions A and B were mixed immediately before the test. A small amount of acetic acid was added to each of the mercaptan samples, and a drop of one of these solutions was added to the mixture of solutions A and B. The result should have been a brown color. No color was obtained even for the 1000 ppm sample. It is possible that the ethanol used as a solvent for the mercaptan interfered with the test.

d. Lead Acetate Test: A saturated solution of lead acetate in dilute acetic acid was prepared. When a mercaptan is added to the lead acetate solution, a yellow precipitate should form. A weak reaction was observed when a solution of 1000 ppm of mercaptan in alcohol was added to the lead acetate solution. No indication was obtained with a 1 ppm solution. In this test, ether was rejected as a mercaptan solvent because it interferes with the precipitation of the yellow salt.

e. Lead Oxide Test: 1.25 g of lead oxide was added to 7.5 g of sodium hydroxide in 40 ml of water. One drop of mercaptan solution was added to two milliliters of the lead oxide solution and shaken in a test tube. Powdered sulfur was then added and the test tube shaken again. Finally, the solution was allowed to stand overnight. When the 1000 ppm mercaptan solution was used in this procedure, the final solution was black, but when the 1 ppm mercaptan solution was used, the final solution gave no visual indication.

It seems evident from these tests that none of them are sufficiently sensitive to detect mercaptans in concentrations associated with small leaks in filters.

Testing N-Amyl Mercaptan for Use with Activated Carbon

Figure 1 is a schematic diagram of the apparatus used in testing the effectiveness of n-amyl mercaptan for detecti'.g leaks in activated carbon beds. This apparatus can be used for other gases or for volatile liquids. It consists of a vacuum pump which pulls air through a carefully prepared and pre-weighed carbon bed that is held in a sintered-glass filtering .ucible. Gas is introduced upstream of the carbon bed, and the flowrate of the air-gas mixture is measured by a rotameter. The rate at which the gas is introduced into the air-stream is measured by a soap-film meter. If the gas is a liquid at room temperature, it can be vaporized by boiling before being passed through the soap-film meter.

N-amyl mercaptan boils at 126 C and therefore condenses in the air at a relatively high temperature. During the tests, this mercaptan condensed on the walls of the glassware through which it flowed, about as soon as it had vaporized. Thus, it was impossible to get enough mercaptan into the air-stream to test the charcoal.

It was also impossible to prevent the mercaptan from leaking from the system, and as a result, the room became contaminated with mercaptan vapor. Powerful ventilating blowers failed to keep the vapor down to an acceptable level. As a result, it was found that the efficiency of personnel is lowered significantly when they are exposed to the mercaptan vapors for more than a few minutes. Thus, it would be undesirable for an occupied personnel area to become contaminated with n-amyl mercaptan vapors, as would happen, for example, if during the course of a test, a leak should be found in a CBR filter installed in a building that was in use.

If a mercaptan is to be used, it will be necessary to use one with a much lower boiling point, such as methyl, ethyl, propyl, or butyl mercaptan, all of which are quite hazardous. (See Table I). Methyl mercaptan is a gas at room temperature and is both flammable and somewhat poisonous. Ethyl mercaptan is a highly explosive liquid. No information could be found on propyl mercaptan, but butyl mercaptan is extremely poisonous. All have exceptionally disagreeable odors.

Because of the disadvantages just discussed, it was decided to reexamine the advisability of using mercaptans for filter testing before continuing with the experiments.

CONCLUSIONS

The problem of developing a field kit to test a collective protector for gas penetration is essentially one of finding a suitable gas and the proper method of detecting that gas. Mercaptans appear too hazardous as a test gas, although the idea of using an odor to test filters may have merit.

Detection of gas penetration requires further investigation. Using people to detect the gas may be practical, but is not as easy to do as it may at first seem. "Spot tests" are apparently not sensitive enough. However, gas chromatography appears to offer a satisfactory method for detecting small quantities of gas.

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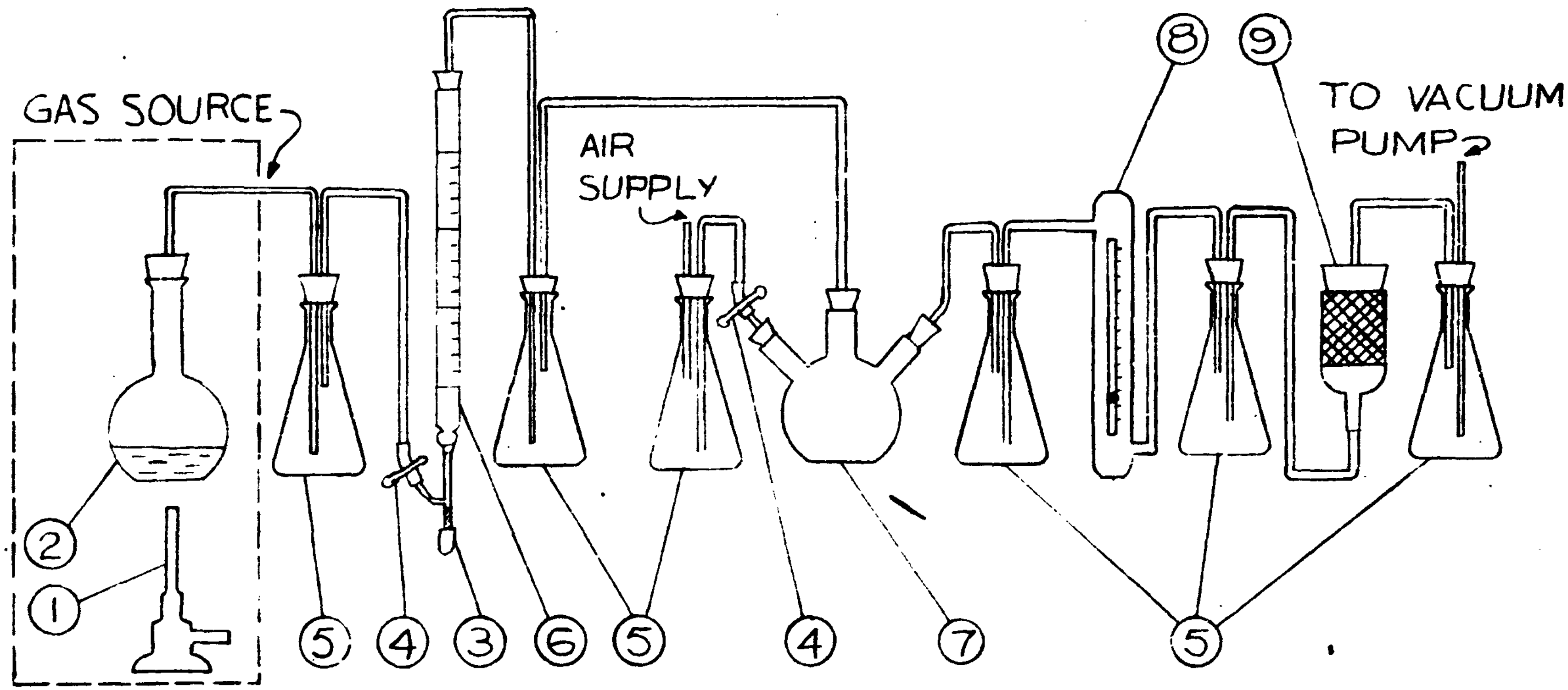

NO.	DESCRIPTION
1	HEAT SOURCE
2	LIQUID SAMPLE VAPORIZER
3	SOAP SUPPLY BULB
4	SCREW CLAMP VALVE
5	TRAP FLASK
6	SOAP FILM METER
7	GAS MIXING CHAMBER
8	FLOWRATOR
9	CARBON BED

CARBON BED
TESTING APPARATUS

FIGURE 1

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3. REPORT TITLE

Investigation of Possible Methods for Testing Gas Filters

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Summary Report, June 1964 to September 1964

5. AUTHOR(S) (Last name, first name, initial)

Gilder, John R.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
16 November 1964	15	11

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO. Y-F011-08-03-301	TN-659
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Activated Carbon	8					
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